



Irradiation embrittlement of pressure vessels steels. IAEA Research Agreement No. 1071/CF

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IRRADIATION EMBRITTLEMENT OF PRESSURE VESSEL
STEELS. IAEA RESEARCH AGREEMENT NO. 1071/CF.

by

A. Nielsen and J. Westermann

Date July 1975

Department or group

Metallurgy

Group's own registration
number(s)

A 178

24 pages + tables + illustrations

Abstract

A Standard Research Programme was approved by the Coordinating Meeting on the 12th of May 1971 of the Working Group covering Engineering Aspects of Irradiation Embrittlement of Pressure Vessel Steels. This Working Group was set up by the International Atomic Energy Agency.

Several institutes in different countries agreed on doing irradiation experiments according to the approved programme on steel A 533B from the American HSST programme.

The Danish contribution covering tensile, impact, and hardness testing of non-irradiated steel and steel irradiated at 290°C to $2 \times 10^{19} \text{ n}_f/\text{cm}^2$ is presented in this report.

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The International Atomic Energy Agency's coordinated programme of research in irradiation embrittlement of pressure vessel steels.
Danish contribution under research agreement No. 1071/CF.

INTRODUCTION

According to a Standard Research Programme approved by the Coordinating Meeting on 12 May 1971 we have carried out tensile and impact testing of testpieces cut from the steel block 03 LK which we received during June 1972 from the Union Carbide Corporation.

One set of testpieces was tested in the unirradiated condition, but given the same heat treatment as irradiated testpieces receive. Another set of testpieces was tested after irradiation at 290°C to 2×10^{19} n/cm².

Two additional blocks, 03 MP and 03 GY, were received and stored for experiments on weld metal.

Material

The steelplate HSBT 05 of quality ASTM A-565 Grade B Class 1 from Lukens has been exhaustively described in report ORNL-TM-5195 by C.E. Childress for Oak Ridge National Laboratory.

Machining

The testpieces have been cut carefully in agreement with the coordinated programme, only the tolerances have been kept closer in order to obtain a satisfactory fit between the testpieces and the irradiation rig.

The impact testpieces have been produced as described in appendix 1 at the end of this report.

As our irradiation rigs have only square holes to accomodate standard impact testpieces the tensile testpieces have been provided with matching steel pieces to conduct gamma heat between testpiece and rig. This arrangement has necessitated slight changes in tolerances of the tensile testpieces.

Irradiation

Reactor Description

The DR 5 reactor at Risø is a 10 MW heavy-water-cooled and -moderated research reactor with highly enriched U-Al fuel elements. The reactor is of a design similar to the British "Pluto" type, and its regular operation at power began in November 1960.

The reactor core consists of 26 fuel elements, each one contains four concentric aluminium-clad fuel tubes, which are arranged to provide a 5 cm centre hole for experiments.

The reactor core and heavy water are contained in an aluminium tank of 200 cm diameter. Vertical and horizontal test holes of 10 and 17.5 cm diameter are located in the radial D₂O reflector.

Outside the aluminium tank is a 30 cm graphite reflector with 10 cm vertical test holes.

Maximum thermal flux	$1.5 \times 10^{14} \text{ n/cm}^2 \text{ sec}$
Maximum fast flux	$4.5 \times 10^{13} \text{ n/cm}^2 \text{ sec}$
Maximum nuclear heat	1.8 W/g Al
Number of fuel elements	26
Length of active zone in a fuel element	61 cm
Fuel enrichment	80% in U^{235}
Average fuel content in the core	2500 g U^{235}
Maximum fuel plate heat flux	82 W/cm^2
Increase in D_2O temperature (along fuel element)	6.6°C
Heavy water outlet temperature	$40 - 45^\circ\text{C}$
Heavy water flow	360 kg/sec
Mean coolant velocity through fuel elements	3.6 m/sec
Thermal power in secondary cooling system	10 MW

Irradiation Rig

The rig is designed for short-time irradiation of structural materials (pressure vessel steels etc.) in the form of full size charpy-V specimens.

The rig is designed to operate in the centre hole of a fuel element in the DR 3 reactor.

The specified temperature of the specimens which are placed in longitudinal holes in two cylindrical aluminium magazines is obtained by means of small built-in electrical heaters. A gas gap between each magazine and the surrounding thimble is used for coarse calibration of the magazine temperature.

The maximum operational temperature is 340°C with the applied magazine material.

The temperature and the temperature distribution in the magazines are measured with three thermocouples in each magazine.

The temperature drop between specimen and magazine is measured by means of two thermocouples placed in the centre of two specimens. The rig atmosphere is helium or a mixture of helium with neon.

Irradiation Procedure

As only one magazine accommodating 12 testpieces was available at the time of irradiation, the 20 testpieces were irradiated during two identical reactor runs and in identical positions.

The reactor reports are enclosed as appendices 3 and 4.

The dose measurement is reported in appendix 5.

It is seen from these reports that:

Tensile specimens 2-17 through 2-20 and impact specimens 2-1 through 2-8 have in reactor run 156 been irradiated for 149.5 hours at $284\text{--}292^{\circ}\text{C}$ to $2.16 \times 10^{19} \text{ n}_f/\text{cm}^2$.

Impact specimens 2-9 through 2-16 have in reactor run 157 been irradiated for 145.7 hours at $283\text{--}292^{\circ}\text{C}$ to $2.03 \times 10^{19} \text{ n}_f/\text{cm}^2$.

Un-irradiated Specimens

In order to evaluate the irradiation damage testpieces 3-1 through 3-20 were prepared identically to those mentioned above, excepting the irradiation. To exclude the effect of the heat treatment during the irradiation experiment these testpieces were heat-treated in a furnace for 158 hours at $288 \pm 5^{\circ}\text{C}$.

Test Results

The tensile testing was done in a lead shielded cell with a 10 ts Instron machine remotely controlled. Special grips were applied which support the testpiece very accurately under the head.

The impact testing was done in a lead shielded cell on a Wolpert PW 15 pendulum with drum brakes and remotely controlled. Available impact energy: 15 kgm.

The hardness testing was done in a lead shielded cell on a Vickers Hardness Tester of the brand "Frankoskop" remotely controlled .
Applied load: 50 kg.

Tensile testing of specimens 3-17 through 3-20 non-irradiated, treated and non-treated.

Code	σ_y N/mm ²	σ_u N/mm ²	δ ^{a)} %	δ_u ^{b)} %	δ_5 ^{c)} %	ψ %
3-17	455	610	21,9	10	23,9	70
3-18	460	615	21,5	11	24,1	62
3-19	460	615	21,7	10	23,8	68
3-20	455	610	23,8	12	26,0	67
Average	457	612	22,2	11	24,4	67

Tensile testing of pieces 2-17 through 2-20, irradiated at $290 \pm 10^\circ\text{C}$ to 2.16×10^{19} or 2.03×10^{19} n_f/cm² ($E_n > 1$ MeV)

Code	σ_y N/mm ²	σ_u N/mm ²	δ ^{a)} %	δ_u ^{b)} %	δ_5 ^{d)} %	ψ %
2-17	555	686	20.8	10	23.3	64
2-18	547	680	20.3	10	22.7	64
2-19	547	680	17.6	9	19.6	61
2-20	550	674	19.6	10	21.9	63
Average	550	680	19.6	10	21.9	63

a) Based on stress-strain curves corrected for elastic effects.

b) Based on maximum of stress-strain curves corrected for elastic effects.

c) Based on gauge length of test piece measured before and after testing.

d) From $\delta_5 = A \frac{x}{5} (\delta_x - \delta_u) + \delta_u$, $x = 32.5/5.0 = 6.5$, $\delta_x = \delta$,

A (from 3-17, 3-18, 3-19, 3-20)

0.94 0.98 0.94 0.95 $A_{av} = 0.95$

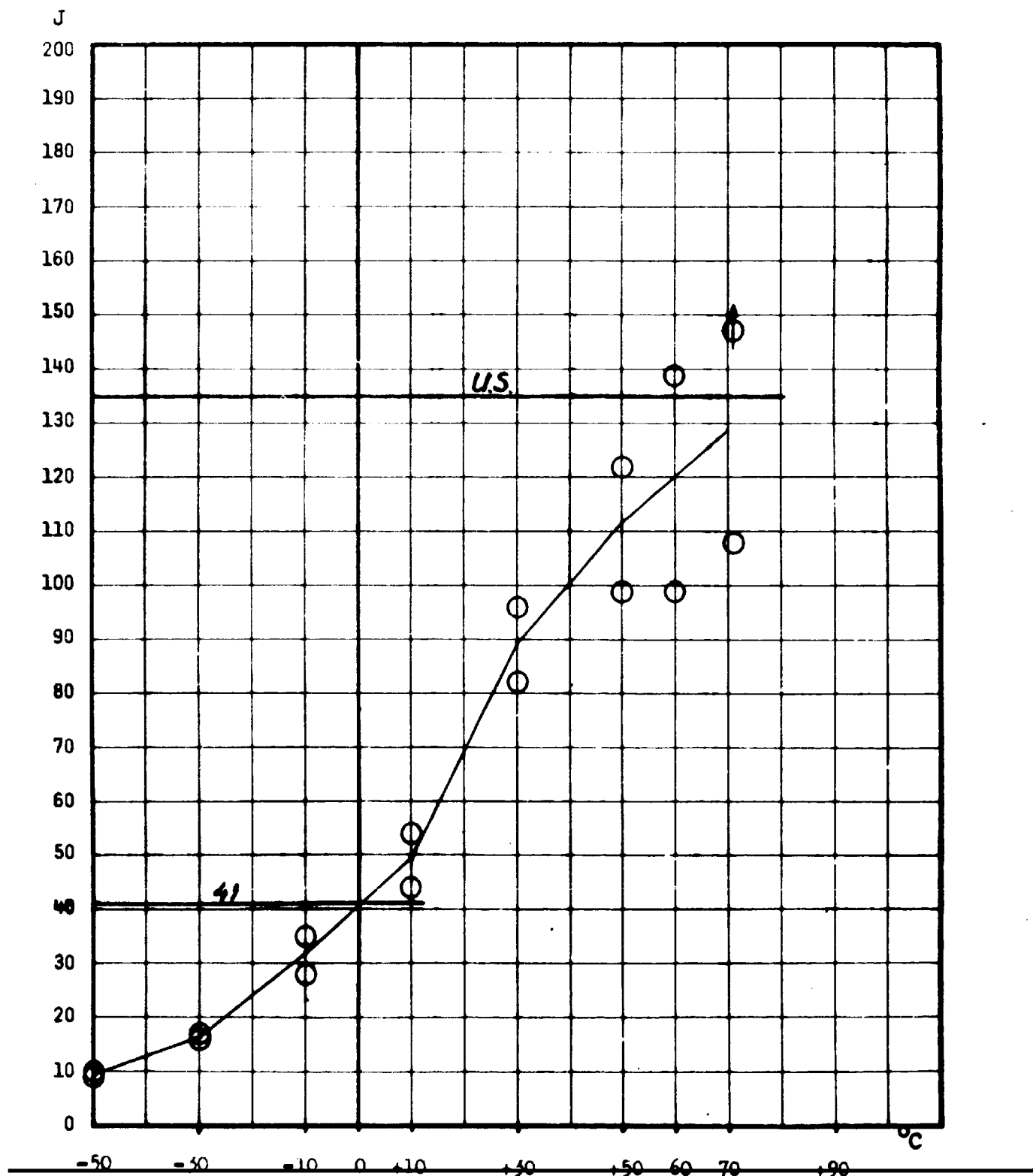
Energy - not in testing of pieces 3-1 through 3-16, non-irradiated, but
 not tested and none at 286 ± 5°C.

<u>Temperature</u> °C	<u>Code</u>	<u>Energy Absorption</u> J
-50	3-1	9
-50	3-14	10
-50	3-8	16
-30	3-16	17
-10	3-2	28
-10	3-15	35
+10	3-9	44
+10	3-3	54
+30	3-10	82
+50	3-4	96
+50	3-11	99
+50	3-5	122
+60	3-6	99
+60	3-12	139 a)
+71	3-7	108 a)
+71	3-13	b) a)

a) Upper shelf energy. No brittle fracture recognizable.

b) Test piece not broken by the max. energy available, i.e. 147 J (15 kgm)

Charpy V-notch testing of pieces 1-1 through 1-16. Non-irradiated,
but heat treated 158 hours at $288 \pm 5^\circ\text{C}$.



Charpy V-notch testing of pieces 2-1 through 2-16, irradiated at
 $290 \pm 10^\circ\text{C}$ to 2.16×10^{19} or 2.03×10^{19} n_f/cm^2 ($E_n > 1\text{MeV}$).

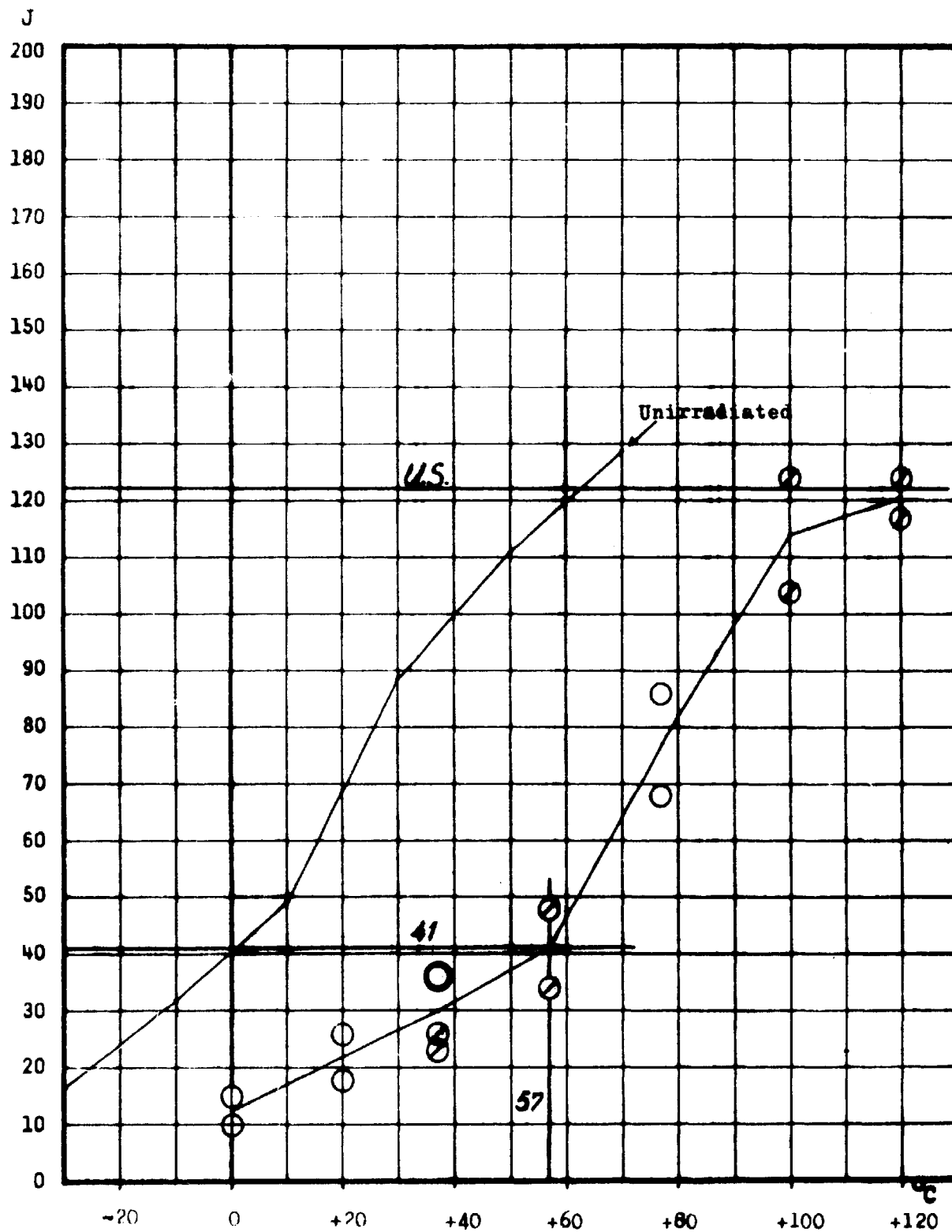
Temperature	Code	Energy absorption
$^\circ\text{C}$		J
0	2-1	10 ^{a)}
0	2-6	15 ^{a)}
20	2-4	18 ^{a)}
20	2-8	26 ^{a)}
37	2-12	23 ^{b)}
37	2-9	26 ^{b)}
37	2-3	36 ^{a)}
37	2-7	36 ^{a)}
57	2-11	34 ^{b)}
57	2-16	48 ^{b)}
77	2-5	68 ^{a)}
77	2-2	86 ^{a)}
100	2-14	104 ^{b)}
100	2-10	124 ^{b) c)}
120	2-13	117 ^{b) c)}
120	2-15	124 ^{b) c)}

a) Irradiated to 2.16×10^{19} n_f/cm^2

b) Irradiated to 2.03×10^{19} n_f/cm^2

c) Upper shelf energy. No brittle fracture recognizable.

Charpy V-notch testing of pieces 2-1 through 2-10, irradiated at
 290 \pm 10°C to 2.16×10^{19} (o) or 2.03×10^{19} (e) n_f/cm^2 ($E_n > 1$ MeV)



Hardness measurement on 3 broken Charpy V-notch pieces, non-irradiated, but heat treated 158 hours at $288 \pm 5^\circ\text{C}$.

Code	H_{V50}^{20} kg/mm ²	H_{V50}^{20} kg/mm ²	H_{V50}^{20} kg/mm ²	H_{V50}^{20} kg/mm ²	Average of the 4 measurements
5-2	202	207	211	196	204
5-8	207	207	203	198	204
5-11	206	211	203	200	205

Average of the three (3) specimens 204

Hardness measurement on 3 broken Charpy V-notch pieces, irradiated at $290 \pm 10^\circ\text{C}$ to $2.16 \times 10^{19} \text{ n}_f/\text{cm}^2$ or $2.03 \times 10^{19} \text{ n}_f/\text{cm}^2$ ($E_n > 1 \text{ MeV}$).

Code	H_{V50}^{20} kg/mm ²	H_{V50}^{20} kg/mm ²	H_{V50}^{20} kg/mm ²	H_{V50}^{20} kg/mm ²	Average of the 4 measurements
2-5	227	229	229	229	229 a)
2-9	239	242	232	232	236 b)
2-11	232	239	247	224	235 b)

Average of the three (3) specimens 233

a) Irradiation : $2.16 \times 10^{19} \text{ n}_f/\text{cm}^2$

b) Irradiation : $2.03 \times 10^{19} \text{ n}_f/\text{cm}^2$

DISCUSSION

From steel block C5LR specimens have been prepared and irradiated at $285 \pm 292^{\circ}\text{C}$ to 2.16 or 2.05×10^{19} n_p/cm^2 . The test results have been compared to results obtained from identical testpieces heat treated at $288 \pm 5^{\circ}\text{C}$, but not irradiated.

The following changes in properties by the irradiation have been observed:

Tensile testing

Yield strength	457 to 550 N/mm^2
Ultimate strength	612 to 680 N/mm^2
Uniform elongation ^{a)}	11 to 10%
Total elongation	22.2 to 19.6%
5 d elongation	24.4 to 21.9%
Lateral contraction ^{a)}	67 to 63%

Impact testing

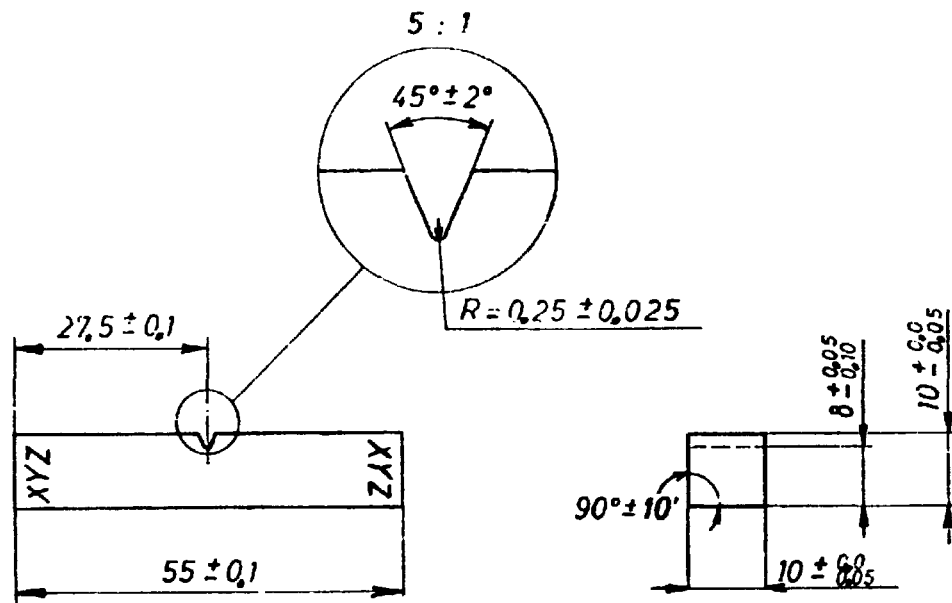
Upper shelf level ^{a)}	(135) to 122 J
Transition temperature at 41 J	0 to $+57^{\circ}\text{C}$

Hardness testing

Vickers Hardness at 30 kg load	2000 to 2300 N/mm^2 (204 to 233 kg/mm^2)
--------------------------------	-------------------------------------------------------------

^{a)} The difference is on a statistical basis not significant.

Charpy V-notch testpiece for irradiation

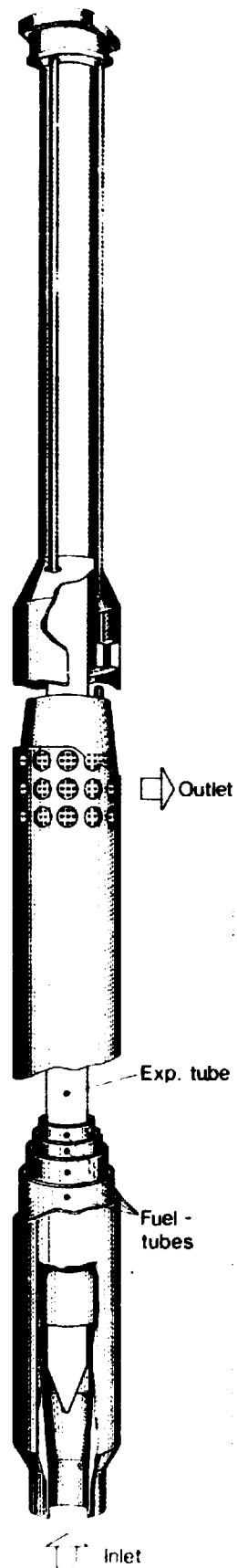


scale 1:1, 5:1

Note

- 1) Machining to be carried out by cutting to 10.2x10.2 mm and grinding to within final tolerances.
- 2) Deviation from linearity: max. 0.030 mm.
- 3) Notch cutting: last but one feed ≤ 0.25 mm
last feed ≤ 0.05 mm
- 4) Numbers to be engraved (not punched) before final grinding.
- 5) End faces to be cleaned of burr.

Mk 4 fuel element



Research Establishment Rise

April 3, 1973

Reactor Department

Internal Report No 580

26 - 6 - 58

Irradiation Report

Project 507

Pressure Vessel Steel Irradiated in

Standard Rig No. 5

Reactor Run 156 (9/5 - 15/5, 1973)

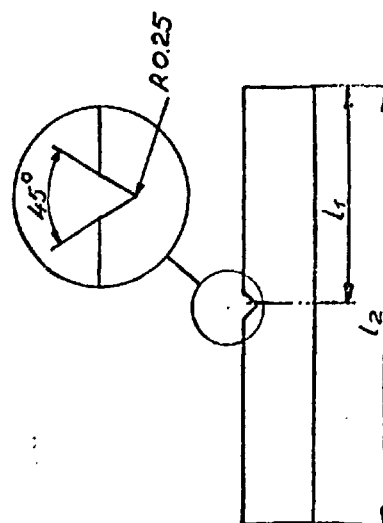
Table 1
Irradiation Data

Reactor run		156
Start of operation		9/3 - 1973
End of operation		15/3 - 1973
Specimens		8 Charpy V-notch 4 tensile see fig. 1, Table 2 and Table 3
Dosimetry		Fe-54 (n.p) Mn-54
Nominal dose	n_f/cm^2	2×10^{19}
Core position		B3
Irradiation time	hours	149,5
Number of trips		See Table 4
Irradiation temperature	$^{\circ}C$	See Table 4

Control of dimensions
Charpy V-notch specimens

Table 2

Test piece no.	l_1 mm	l_2 mm	b_1 mm	b_2 mm	d mm	Linearity	Notch	Comments
1	27.5	55.0	9.96	9.97		o.k.	o.k.	
2	27.5	55.0	9.96	9.97		o.k.	o.k.	
3	27.5	55.0	9.96	9.97		o.k.	o.k.	
4	27.5	55.0	9.96	9.97		o.k.	o.k.	
5	27.5	55.0	9.965	9.975		o.k.	o.k.	
6	27.5	55.0	9.96	9.975		o.k.	o.k.	
7	27.5	55.0	9.96	9.975		o.k.	o.k.	
8	27.5	55.0	9.96	9.97		o.k.	o.k.	



Specifications

l_1 mm	l_2 mm	b_1 mm	b_2 mm	d mm
27.5 ± 0.1	55 ± 0.1	10 ± 0	10 ± 0	2 ± 0.1

Control of dimensions

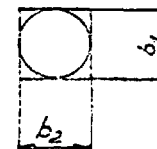
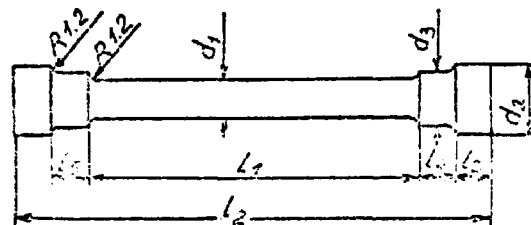
Tensile specimens

Table 3

Test piece no.	d ₁ mm	d ₂ mm	d ₃ mm	l ₁ mm	l ₂ mm	l ₃ mm	l ₄	l ₅	Adaptors		Linearity	Comments
									b ₁ mm	b ₂ mm		
20	5.0	9.98	6.45	34.65	55.05	5.0	5.3	4.9	9.99	9.98	o.k.	
17	5.0	9.98	6.45	34.9	55.0	5.0	5.0	5.0	9.98	9.98	o.k.	
18	5.0	9.98	6.45	34.9	55.0	5.0	5.1	5.0	9.98	9.98	o.k.	
19	5.0	9.97	6.45	34.9	55.0	5.0	5.0	5.0	9.97	9.97	o.k.	

Specifications

d ₁ mm	d ₂ mm	d ₃ mm	l ₁ mm	l ₂ mm	l ₃ mm	l ₄ , l ₅ mm	b ₁	b ₂
5 ^{±0.01}	10 ^{±0.1}	6.5 ^{±0.1}	35 ^{±0.1}	55 ^{±0.1}	5	5	10 ⁺⁰ -0.05	10 ⁺⁰ -0.05



Temperature of test pieces

Table 4

Date	9/3	10/3	11/3	12/3	13/3	14/3	15/3	Comments
^x Average temp. for upper part of magazine	285	286	286	285	284	285	286	
^x Average temp. for lower part of magazine	291	292	292	291	290	291	292	

^xThe average temp. is calculated for each day from observations made each second hour. The variation in temperature is $\pm 3^{\circ}\text{C}$.

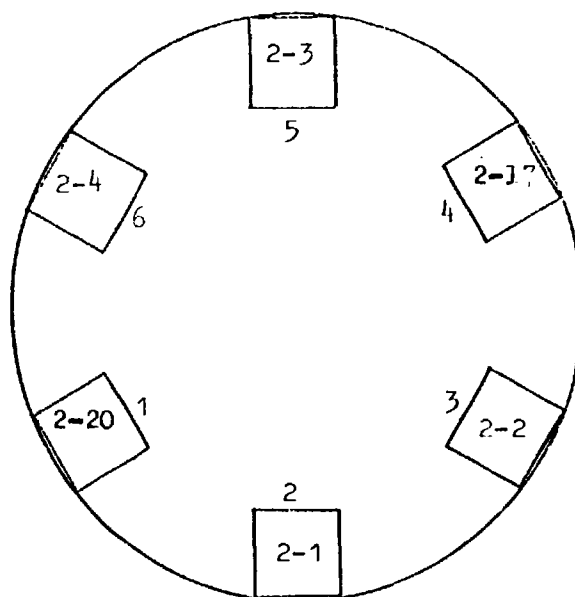
Interruptions of service:

1. Reactortrips: 9/3-73 shut down 13⁵⁰ Full power 15⁵⁵
 15/3-73 shut down 03⁵⁷ Full power 04⁴⁶
 15/3-73 shut down 07⁴⁴ Full power 08³¹

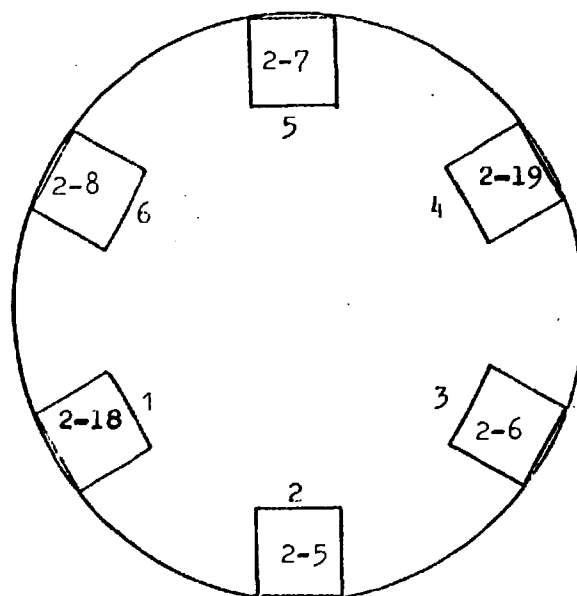
2. Temp. decrease: 15/3-73 08³¹ - 10⁴⁰ $T_{\text{upper part}} = 272^{\circ}\text{C}$
 $T_{\text{lower part}} = 278^{\circ}\text{C}$

Fig. 1.

Loading of standard rig No. 5



Upper half of magazine



Lower half of magazine

Research Establishment Rise

May 10, 1973

Reactor Department

26 - 6 - 10

Irradiation Report

Project 507

Pressure Vessel Steel Irradiated in

Standard Rig No. 5

Reactor Run 157 (6/4 - 12/4, 1973)

Table 1

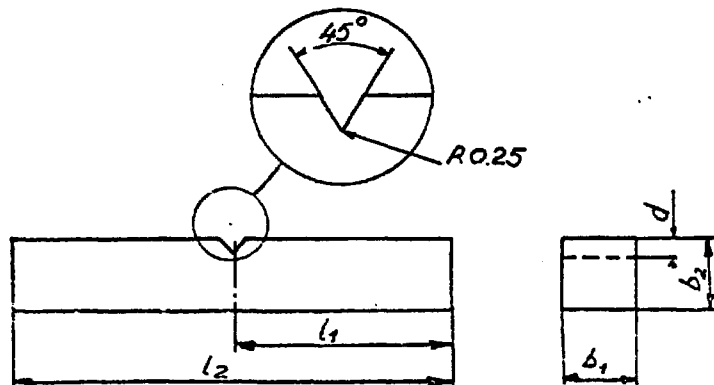
Irradiation Data

Reactor run		157
Start of operation		6/4 - 1973
End of operation		12/4 - 1973
Specimens		8 Charpy V See fig. 1 Table 2
Dosimetry		Fe-54 (n.p) Mn-54
Nominal Dose	n_f/cm^2	2×10^{19}
Core position		B3
Irradiation time	hours	145.7
Number of trips		See Table 3
Irradiation temp.	$^{\circ}C$	See Table 3

Control of dimensions
Charpy V-notch specimens

Table 2

Test piece No.	l_1 mm	l_2 mm	b_1 mm	b_2 mm	d mm	Linearity	Notch	Comments
2 - 9	27.5	55.0	9.97	9.97		o.k.	o.k.	
2 - 10	27.5	55.0	9.97	9.98		o.k.	o.k.	
2 - 11	27.5	55.0	9.96	9.97		o.k.	o.k.	
2 - 12	27.5	54.9	9.96	9.97		o.k.	o.k.	
2 - 13	27.5	55.0	9.96	9.97		o.k.	o.k.	
2 - 14	27.5	55.0	9.96	9.98		o.k.	o.k.	
2 - 15	27.5	55.0	9.97	9.97		o.k.	o.k.	
2 - 16	27.5	55.0	9.97	9.97		o.k.	o.k.	



Specifications

l_1 mm	l_2 mm	b_1 mm	b_2 mm	d mm
27.5 ± 0.1	55 ± 0.1	$10^{+0}_{-0.05}$	$10^{+0}_{-0.05}$	2 ± 0.1

Temperature of test pieces in magazine C

Table 3

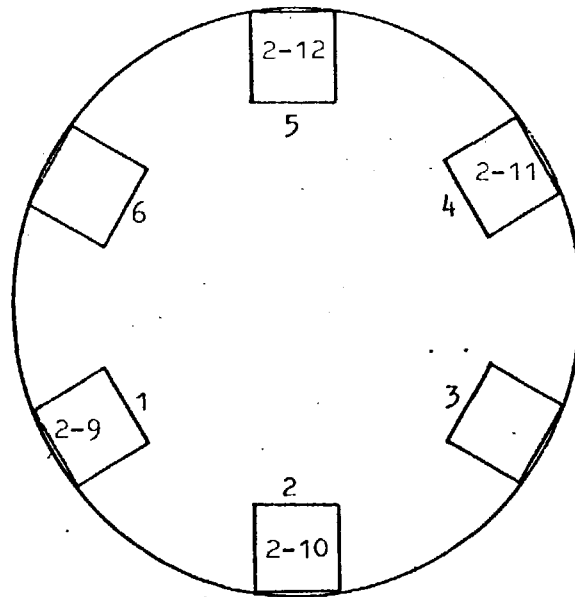
Date	6/4	7/4	8/4	9/4	10/4	11/4	12/4	Comments
^x Average temp. for upper part of magazine	284	284	284	284	284	283	284	
^x Average temp. for lower part of magazine	292	292	292	292	292	291	292	

^xThe average temp. is calculated for each day from observations made each second hour. The variation in temperature is $\pm 3^{\circ}\text{C}$.

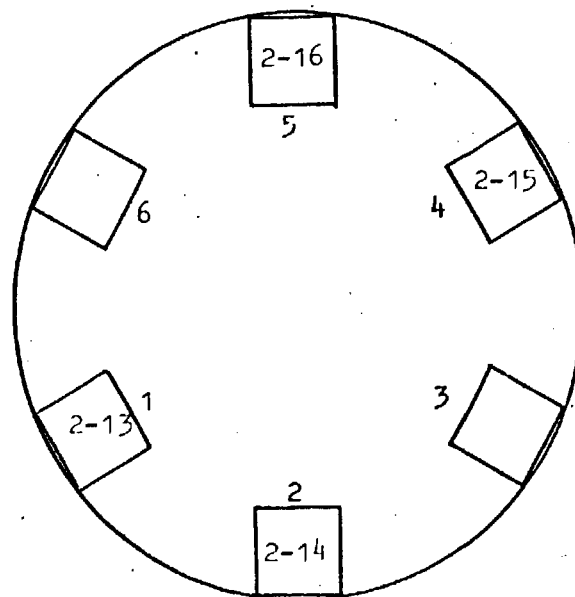
Interruptions of service: None.

Fig. 1

Loading of standard rig No. 5



Upper half of magazine



Lower half of magazine

Reactor Physics Department
DR 1

DR 1. 4 July 1975.

Thermal and Fast Neutron Flux in Steel

Reactor period	Test sample	Irradiation time, hrs.	Weight g	Fast flux n/cm ² /sec	Thermal flux n/cm ² /sec
156	2 - 1	149.5	1.160	4.02×10^{13}	5.56×10^{13}
157	2 - 16	145.7	0.465	3.88×10^{13}	5.93×10^{13}

The fast flux is measured by means of the threshold reaction Fe-54(m,p)Mn-54. The thermal flux is measured by means of the reaction Fe-58(n, γ)Fe-59.